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MOLYBDENUM ALLOY X-RAY TARGETS HAVING  
UNIFORM GRAIN STRUCTURE

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BACKGROUND OF THE INVENTION

X-ray producing devices are extremely valuable tools that are used in a wide variety of applications, both industrial and medical. For example, such equipment is commonly used in areas such as diagnostic and  
10 therapeutic radiology; semiconductor manufacture and fabrication; and materials analysis and testing.

While used in a number of different applications, the basic structure and operation of x-ray devices is similar. X-rays are produced when electrons are generated, accelerated to a high speed, and then stopped  
15 abruptly. Typically, this entire process takes place within a vacuum formed x-ray generating tube. An x-ray tube ordinarily includes three primary elements: a cathode assembly, which is the source of electrons; an anode, which is axially spaced apart from the cathode and oriented so as to receive electrons emitted by the cathode; and some mechanism for  
20 applying a high voltage for driving the electrons from the cathode to the anode. Usually, the cathode assembly is composed of a metallic cathode head having a cathode cup. Disposed within the cathode cup is a filament that, when heated via an electrical current, emits electrons.

The three x-ray tube elements are usually positioned within an  
25 evacuated glass tube and connected within an electrical circuit. The electrical circuit is connected so that the voltage generation element can apply a very high voltage (ranging from about ten thousand to in excess of hundreds of thousands of volts) between the anode and the cathode. This high voltage differential causes the electrons that are emitted from the  
30 cathode filament to accelerate at a very high velocity towards an x-ray "target" positioned on the anode in the form of a thin stream, or beam. The x-ray target includes a plate with a focal track. When the electrons strike

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the target surface, the kinetic energy of the striking electron beam is converted to electromagnetic waves of very high frequency, i.e., x-rays. The resulting x-rays emanate from the anode target surface, and are then collimated through a window formed in the x-ray device for penetration into an object, such as an area of a patient's body.

Unfortunately, known processes for making plates have limitations that have prevented artisans from develop a simple effective process for quickly effectively making plates useful in x-ray targets. A particularly limiting disadvantage of known single forging processes, for instance, is that such processes are wasteful and inefficient, making them costly and impractical to implement. Known processes make products have poor uniform grain size. Poor uniformity in grain size interferes with the performance of X-ray anodes. Also, known processes produce anodes having poor temperature properties.

For the foregoing reasons, there is a need to develop a process that produces x-ray targets having uniform properties.

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## SUMMARY OF THE INVENTION

The invention relates to a process for making a cross-directionally worked molybdenum plate. The plate is preferably used in an X-ray target so that the plate functions as an anode. The process involves (a) reducing ammonium molybdate and forming molybdenum metal powder; (b) consolidating a molybdenum component made of molybdenum metal powder and an alloying element to a first workpiece, the alloying element being selected from the group consisting of titanium, zirconium, hafnium, carbon, lanthanum oxide, and combinations thereof; (c) thermally treating the first workpiece and subjecting the workpiece to thermo-mechanical forces in a first direction, and thereby forming a second workpiece; (d)

thermally treating the second workpiece and subjecting the second workpiece to thermo-mechanical forces in a second direction that is different from the first direction; (e) subjecting the thermomechanically treated second workpiece to a recrystallization heat treatment step, and  
5 thereby forming a heat-treated cross-directionally worked workpiece; and (f) subjecting the heat-treated, cross-directionally worked workpiece to a slicing step or a machining step, and thereby forming the cross-directionally worked molybdenum plate.

In one embodiment, the invention relates to a member made by  
10 such a process, in which the member includes a plate and a stem attached to the plate.

In one embodiment, the invention relates to a plate involving a cross-directionally worked molybdenum component selected from the group consisting of (i) a molybdenum component containing molybdenum  
15 and an alloying element selected from the group consisting of titanium, zirconium, hafnium, carbon, lanthanum oxide, and combinations thereof or (ii) a molybdenum component of molybdenum, niobium and an alloying element selected from the group consisting of titanium, zirconium, hafnium, carbon, lanthanum oxide, and combinations thereof or (iii) a  
20 molybdenum component of molybdenum, tungsten in an amount ranging from about 1 to about 30 wt.% and an alloying element selected from the group consisting of titanium, zirconium, hafnium, carbon, lanthanum oxide, and combinations thereof; in which the plate has a radial strength of at least about 60 ksi when the plate is exposed to a temperature of about  
25 1600°C.

In another embodiment, the invention relates to an X-ray target: (a) a plate involving a cross-directionally worked molybdenum component selected from the group consisting of (i) a molybdenum component containing molybdenum and an alloying element selected from the group  
30 consisting of titanium, zirconium, hafnium, carbon, lanthanum oxide, and

combinations thereof or (ii) a molybdenum component including molybdenum, niobium and an alloying element selected from the group consisting of titanium, zirconium, hafnium, carbon, lanthanum oxide, and combinations thereof or (iii) a molybdenum component including  
5 molybdenum, tungsten in an amount ranging from about 1 to about 30 wt.% and an alloying element selected from the group consisting of titanium, zirconium, hafnium, carbon, lanthanum oxide, and combinations thereof; in which the plate has a radial strength of at least about 60 ksi when the plate is exposed to a temperature of about 1600°C; (b) a focal  
10 track located on a surface of the plate; and (c) a stem extending from the plate.

#### DESCRIPTION OF THE FIGURES

These and other features, aspects, and advantages of the present  
15 invention will become better understood with reference to the following description and appended claims, where:

Fig. 1 is a schematic showing how a billet/ingot can be extruded in accordance to the invention;

Figs. 2 (a) and 2 (b) are a schematic showing how the first  
20 workpiece can be upset forged in accordance to the invention;

Fig. 3(a) is a view of the second workpiece generated during the upset forging operation with removal of centering disk affected material at A;

Fig. 3(b) is a view of the second workpiece formed from upset  
25 forging and a plate that can be sliced from the billet;

Fig. 4 is a side view showing a workpiece being subjected to hammer forging;

Fig. 5 shows the second workpiece formed from a workpiece that is subjected to hammer forging; and  
30

Fig. 6 is a side view of a stem that can be attached to the plate made in accordance to the invention.

### DESCRIPTION OF THE INVENTION

5       The cross-directionally worked molybdenum plate is generally made by a process that (a) reduces ammonium molybdate and forming molybdenum metal powder; (b) consolidates a molybdenum component made of molybdenum metal powder and an alloying element to a first workpiece, the alloying element being selected from the group consisting  
10 of titanium, zirconium, hafnium, carbon, lanthanum oxide, and combinations thereof; (c) thermally treats the first workpiece and subjecting the workpiece to thermo-mechanical forces in a first direction, and thereby forming a second workpiece; (d) thermally treats the second workpiece and subjecting the second workpiece to thermo-mechanical  
15 forces in a second direction that is different from the first direction; (e) subjects the thermomechanically treated second workpiece to a recrystallization heat treatment step, and thereby forming a heat-treated cross-directionally worked workpiece; and (f) subjects the heat-treated, cross-directionally worked workpiece to a slicing step or a machining step,  
20 and thereby forms the cross-directionally worked molybdenum plate.

      The molybdenum metal powder is made by reducing ammonium molybdate by any suitable process. Generally, this is done through a conventional hydrogen reduction process. The process consolidates a molybdenum component including molybdenum metal powder and an  
25 alloying element to a first workpiece. In one embodiment, the molybdenum component is consolidated into the first workpiece by a powder metallurgical technique. Such a process generally involves the steps of cold isostatic pressing of the molybdenum metal powder and any alloying components into a "green" preform of the first workpiece. In another embodiment, the molybdenum component is consolidated into  
30 the first workpiece by a melt casting technique. Such a process may involve vacuum arc casting, ebeam melting, or plasma arc casting of an ingot. The alloying element is

generally selected from the group consisting of titanium, zirconium, hafnium, carbon, lanthanum oxide, and combinations thereof. In one embodiment, the first workpiece further includes niobium in an amount that is less than about 3 wt.%, preferably from about 1 wt % to about 3 wt.%. In  
5 another embodiment, the alloying element is present in an amount that is about 1.2 wt. %, or less. In another embodiment, the alloying element is present in an amount ranging from about 1 wt.% to about 1.5 wt.%. In another embodiment, the first workpiece further includes tungsten in an amount ranging from about 1 to about 30 wt.%.

10 The first workpiece is thermally treated and subjected to thermo-mechanical forces in a first direction, so that a second workpiece forms. The thermal treatment of the first workpiece is generally done at a temperature that is at least about 2100°F (about 1148°C) and preferably from about 2500°F (about 1371°C) to about 3000 °F (1648°C).

15 The thermally treated first workpiece is subjected to any thermomechanical treatment, which when practiced in accordance to the invention, produces a plate that is useful in an X-ray target. In one embodiment, the first workpiece is a billet or an ingot and the first workpiece is thermo-mechanically treated by extruding the billet or the  
20 ingot to a ratio of reduction ( $D_o:D_f$ ) in a cross-sectional area ranging from about 3:1 through about 4:1. More particularly, referring to Fig. 1, the billet/ingot is extruded along length "L" to a suitable ratio of reduction ( $D_o:D_f$ ). The billet length can be vary and preferably is from about 14" (about 36 cm) to about 32" (about 82 cm).

25 The second workpiece is then thermally treated and subjected to thermo-mechanical forces in a second direction that is different from the first direction. In one embodiment, the second workpiece is subjected to upset forging. The thermal treatment of the first workpiece is generally done at a temperature that is at least about 2100°F (about 1148°C) and  
30 preferably from about 2500°F (about 1371°C) to about 3000 °F (1648°C).

The thermally treated second workpiece is subjected to any thermo-mechanical treatment, which when practiced in accordance to the invention, produces a plate that is useful in an X-ray target. In one embodiment, the second workpiece is upset forged by a closed die forging process with a closed die that is dimensioned to form a plate. In another  
5 embodiment, the second workpiece is upset forged by an open die (pancake) forging process with an open die that is dimensioned to form a plate. In another embodiment, the closed die is further dimensioned to include a mold for a stem (shown in Fig. 6) so that the plate formed by the  
10 process further includes a stem. As such, the invention encompasses a member made by the process of the invention, in which the member has a plate and a stem attached to the plate.

Referring to Figs. 2(a) and 2(b), Fig. 2(a) shows the first workpiece (10) at the start of the upset forging process in which the workpiece is  
15 placed in the press liner (12), between centering disks (14,16) and within an extrusion liner (19), such the moving centering disk (14) applies a main ram tonnage (18) against the stationary centering disk (16).

Fig. 2(b) depicts the end of the forming process in which the workpiece (10) has been subjected to thermo-mechanical forces thereby  
20 forming a forged workpiece (or forged billet (20)).

In another embodiment, as shown in Figs. 4 and 5, the second workpiece is subjected to hammer-forging instead of upset forging. Fig. 4 shows a workpiece being subjected to hammer forging, and Fig. 5 shows the second workpiece formed from the first workpiece. In this embodiment,  
25 the ratio of  $D_o$  cross-sectional area to  $D_f$  cross-sectional area preferably ranges from 1:2.0 to 1:2.8.

The thermo-mechanically treated second workpiece is subjected to a recrystallization heat treatment step to form a fully annealed, cross-directionally worked workpiece. This is generally done by through  
30 conventional heat treatment methods.

The heat treatment is generally done at a temperature that is at least about 2100°F (about 1148°C) and preferably from about 2500°F (about 1371°C) to about 3000 °F (1648°C).

5 The heat-treated, cross-directionally worked workpiece is then subjected to a slicing step or a machining step, and thereby the cross-directionally worked molybdenum plate forms. The material that does not form the billet during upset forging (the "affected material") is removed by the centering disks (CD), as shown in Fig 3(a) and 4. A plate can be sliced from the billet per the orientation shown in the Fig. 3(b) Advantageously, 10 the entire billet is useful for making plates.

The plate made by the process of the invention has any suitable dimension that enables the plate to be used in a sputtering target. In one preferred embodiment, the plate has a diameter ranging from about 6" (about 15 cm) to about 14 " (about 36 cm) and a thickness/height ranging 15 from about 0.5" (about 1 cm) to about 2" (about 5 cm). In another embodiment, the plate has a diameter ranging from about 1" (about 2.54 cm) to about 14" (about 36 cm) and a thickness/ height ranging from about 1/4" (about 0.6 cm) to about 7" (about 18 cm).

Advantageously, the plate made in accordance to the invention has 20 high temperature properties. In one embodiment, the plate has a radial strength of at least about 60 ksi when the plate is exposed to a temperature of about 1600°C. When the alloying element includes lanthanum oxide, the plate made by the process of the invention has improved creep resistance, as compared to a plate made without 25 lanthanum oxide. In one embodiment the plate further includes a stem.

As such, in a preferred embodiment of the invention, the invention provides a plate having a cross-directionally worked molybdenum component selected from the group consisting of (i) a molybdenum component containing molybdenum and an alloying element selected from 30 the group consisting of titanium, zirconium, hafnium, carbon, lanthanum



oxide, and combinations thereof or (ii) a molybdenum component including molybdenum, niobium and an alloying element selected from the group consisting of titanium, zirconium, hafnium, carbon, lanthanum oxide, and combinations thereof or (iii) a molybdenum component made of  
5 molybdenum, tungsten in an amount ranging from about 1 to about 30 wt.% and an alloying element selected from the group consisting of titanium, zirconium, hafnium, carbon, lanthanum oxide, and combinations thereof; in which the plate has a radial strength of at least about 60 ksi when the plate is exposed to a temperature of about 1600°C.

10 In one embodiment, the stem also includes a cross-directionally worked molybdenum component selected from the group consisting of (i) a molybdenum component containing molybdenum and an alloying element selected from the group consisting of titanium, zirconium, hafnium, carbon, lanthanum oxide, and combinations thereof or (ii) a molybdenum  
15 component involving molybdenum, niobium and an alloying element selected from the group consisting of titanium, zirconium, hafnium, carbon, lanthanum oxide, and combinations thereof or (iii) a molybdenum component including molybdenum, tungsten in an amount ranging from about 1 to about 30 wt.% and an alloying element selected from the group  
20 consisting of titanium, zirconium, hafnium, carbon, lanthanum oxide, and combinations thereof, in which the stem also has a strength of at least about 60 ksi when the stem is exposed to a temperature of about 1600°C.

Although the invention has been described in detail in the foregoing for the purpose of illustration, it is to be understood that such detail is solely  
25 for that purpose and that variations can be made therein by those skilled in the art without departing from the spirit and scope of the invention except as it may be limited by the claims.